Note

Intergranular Fracture in Fe-Mn Austenitic Alloys at 77 K

1. Introduction

Intergranular fracture has been frequently observed at low temperatures in high Mn austenitic steels being associated with ductile-brittle transition (DBT).¹⁻³⁾ Several reasons concerning this embrittlement have been pointed out so far. The main ideas are (a) intrinsic properties of high Mn steels,^{3,4)} (b) grain boundary (G.B.) segregation of impurity elements,^{5,6)} and (c) stress concentration at G.B. resulting from inhomogeneous plastic flow in the interior of grains.^{4,7,8)} Reasons (a) and (b) are closely related each other since they commonly emphasize the importance of the strength of G.B. Reason (c) is also dependent upon the G.B. strength concerning whether it becomes a main controlling factor or minor one for the occurrence of G.B. cracking. Therefore, the features of plastic flow and the G.B. strength have to be discussed simultaneously.

The intergranular fracture in Fe-Mn binary austenitic alloys were found earlier by Russian workers^{2,3)} and then studied by Matsuura *et al.*,⁹⁾ but the detail of the fracture behaviour has not been reported. Recently, Tomota *et al.* have studied Fe-36wt%Mn alloy and observed inhomogeneous plastic flow.⁷⁾ They also observed Mn enrichment around the G.B. by a scanning Auger electron spectroscopy (AES) but could not catch any evidence concerning the G.B. segregation of other elements. Subsequently, Nagai and Morris showed fractographs by a scanning electron microscopy (SEM) where sharp steps resulting from concentrated plastic flow were recognized only at one side of the separated grain boundary.⁸⁾ This suggests reason (c) is reasonable for this case. It also explains another experimental fact that DBT temperature was decreased by grain refining in an Fe-40wt%Mn alloy.⁹⁾

Quite recently, however, cooling rate after solution treatment has been found to influence the DBT temperature^{5,6,10)} by impact tests. Xue *et al.*⁵⁾ observed that the degree of Mn enrichment at G.B. was changed by the cooling rate or solutionizing condition and suggested that Mn enriched zone along grain boundary might affect the plastic deformation behaviour. Contrary to this, Strum and Morris⁶⁾ re-

ported that G.B. segregation of B was detected in a slowly cooled specimen while N in a rapidly quenched one by AES, although their alloys were containing Cr and N (not binary alloys). Furthermore, they have suspected the data on Mn segregation because they obtained similar Mn intensity profiles resulting from Ar⁺ sputtering at transgranularly fractured area as well as at intergranularly fractured one in an identical specimen. There seems to exist some difficulties in precisely measuring G.B. the chemistry by AES. On the other hand, Kondo *et al.*¹⁰⁾ have also found the effect of cooling rate on fracture mode independently of above workers. They are obtaining very promising evidence for B segregation during cooling by using an alpha particle fission track etching (FTE).¹¹⁾ The true reason of intergranular fracture in high Mn binary austenite is then now in confusion. The aim of this note is to offer some observations on tensile deformation and fracture behaviour of Fe-high Mn austenitic alloys and consequently to emphasize that the G.B. strength of Fe-Mn binary austenitic alloys is firstly estimated to be intrinsically weak.

2. Experimental Procedure

The chemical compositions of the alloys used in this experiment are listed in Table 1. They are denoted as 40 Mn, 44 Mn, and 42Mn-5Cr according to their main chemical compositions. 40 Mn was made by induction melting using electrolytic iron (purity: 99.9 %) and manganese (99.9 %). As a reference alloy, more pure binary alloy, 44 Mn was used. This alloy was examined already in Ref. 10) and kindly supplied by Prof. Shibata of The University of Tokyo. Since most of commercially developed steels for low temperature use contain Cr, C and/or N, 42Mn-5Cr produced by Sanyo Special Steel Co., Ltd. was added for another reference alloy. A series of investigation by Tomota *et al.*^{7,12,13)} have suggested that Cr addition is considered to be effective in preventing the intergranular fracture but N and C are not. After homogenizing at 1 473 K for 86.4 ks, they were hot-forged and then cold-rolled to sheets with a thickness of 1.5 mm, from which plate tension specimens with a gage length of 30 mm and a cross section of 1.5 mm by 4 mm were made. Three kinds of solution treatments listed in Table 2 were given to the specimens in order to change the grain size. After the solution treatment, the specimens were cooled in

Table 1. Chemical compositions of specimens. (wt%)

Specimen	C	Si	Mn	Cr	P	S	Ni	Al	N
40 Mn	0.019	0.01	39.55	—	0.011	0.019	—	—	—
44 Mn	0.004	0.017	44.4	—	0.002	0.019	—	0.005	0.002
42Mn-5Cr	0.16	0.55	41.50	5.04	0.022	0.022	0.04	0.001	0.029

furnace or quenched into water. All the heat treatments were conducted in an argon gas atmosphere. The ASTM grain size was determined from optical micrographs and the results are presented in Table 2 where the values in parentheses are for the furnace-cooled specimens. Henceforce, they are called as SGS (small grain size), MGS (middle grain size), and LGS (large grain size), respectively, as indicated in Table 2. Tension tests were carried out on a gear-driven type machine in liquid nitrogen (at 77 K) at an initial strain rate of $2.8 \times 10^{-3}/s$. The percentage of intergranular fracture was measured in the central region of the fractured surface by using a point counting method.

3. Experimental Results and Discussion

A stress-strain curve and the aspect of fractured surface for 40 Mn with MGS are shown in Figs. 1 and 2, respectively. The central region of the fractured surface consists mainly of intergranular fracture (Fig. 1(a)) while the percentage of dimple area increases as travelling toward the free surface (b). A

Table 2. Relationship between grain size (μm) and solution treatment condition.

Specimen	Heat		
	1 323 K, 1 h SGS	1 423 K, 6 h MGS	1 473 K, 22 h LGS
40 Mn	73 (76)	105 (124)	161 (163)
44 Mn	81 (83)	143 (148)	164 (183)
42Mn-5Cr	64 (74)	74 (84)	109 (112)

Values in parentheses are for furnace-cooled specimens.

Solid lines: The water-quenched specimens (W.Q.)
Dashed lines: The furnace-cooled specimens (F.C.)

Fig. 1. Nominal stress-strain curves for the MGS specimens in 40 Mn, 44 Mn, and 42Mn-5Cr at 77 K.

little amount of necking was observed, so that the G.B. decohesion seemed to occur under the aid of triaxial stress after a considerable amount of plastic deformation.

The effect of grain size on fractography is summarized in Table 3 where intergranular fracture was found to be suppressed by grain refining. The intergranularly fractured area like Fig. 2(a) was increased with an increase in grain size. In the LGS specimens, G.B. cracking was observed even at the free surface as shown in Fig. 3(a). Here, each grain seemed to be well plastically deformed, so that the microcrack was estimated to be yielded somewhat in a ductile manner rather than in a brittle one. When an MGS fractured specimen was sectioned longitudinally and polished, similar subcracks were found in its central region. The intergranular fracture process in this alloy is thus a little different from other ones in lower Mn alloys such as ferritic binary steels,^{7,12,14,15} the so-called Hadfield steel (approximately 12wt%Mn-1.0wt%C),^{4,16} and age-hardened Fe-Mn-Ni alloys.¹⁷ Particularly in the latter two kinds of steels, the stress concentration at the G.B. due to heterogeneous plastic flow is much more distinguished than this case. For comparison, the aspect of surface of plastically deformed Hadfield steel¹² is presented in Fig. 3(b). Although the intergranular fracture is grain size dependent and heterogeneous flow was observed in some degree in 40 Mn, the stress concentration caused by slip looks not so intense (for instance, see Fig. 3(a)).

When solutionized specimens were cooled in furnace, the percentage of intergranular fracture was found to be slightly decreased in the MGS specimens (see Fig. 2(c) and Table 3).

Table 3. Percentage of intergranular fracture at 77 K. (%)

Specimen		WQ	FC
40 Mn	SGS	6	4
	MGS	90	73
	LGS	99	99
44 Mn	SGS	17	3
	MGS	93	15
	LGS	97	93
42Mn-5Cr	SGS	0	0
	MGS	0	0
	LGS	0	70

(a) The water-quenched MGS specimen (central region of the fractured surface)
(b) The water-quenched MGS specimen (edge region)
(c) The furnace-cooled MGS specimen (central region)

Fig. 2. Aspects of fractured surfaces in 40 Mn.

Fig. 3. Grain boundary cracking observed on the surface of 40 Mn specimen with LGS in tension (a) and Hadfield steel in compression (b)¹²⁾ at 77 K. Load direction is horizontal of the micrographs.

Therefore, 44 Mn, in which a large cooling rate dependence of DBT was already reported by Kondo *et al.*,¹⁰⁾ was examined in the same way as 40 Mn. As results, the influence of cooling rate was revealed to be more pronounced. In the MGS specimens, fractography changed from intergranular dominant pattern in the water quenched specimen to dimple one in the furnace-cooled one (see Table 3). However, in the SGS or LGS specimens, the influence of cooling rate is not so large. The grain refining is effective in preventing the intergranular fracture as well as in 40 Mn. The aspects of free surface of deformed specimens in the water-quenched and furnace-cooled specimens were compared by optical microscopy, but no reliable evidence for a change in deformation manner could be found out, whereas their fracture mode was drastically changed from intergranular to dimpled transgranular. No significant difference can be recognized in stress-strain curves in Fig. 1 (compare solid and dashed curves). The strain hardening exponent (n -value) is almost identical: *i.e.*, approximately 0.5 ± 0.03 in both cases. Therefore, it is difficult to attribute an origin of the cooling rate effect to a change in deformation manner. It seems to be more promising to examine the segregation behaviour of B during the cooling as suggested by Shibata *et al.*¹¹⁾ Since B was not added with a special intention and was believed to be entered from raw material and/or crucible, the subtle difference between 40 Mn and 44 Mn described above would possibly occur if B segregation were true. A little stress concentration seems to be sufficient to induce the G.B. decohesion in these alloys, while intense stress concentration resulting from remarkable inhomogeneous plastic flow or matrix toughening against cleavage fracture must be necessary in lower Mn steels (whose microstructures are lath martensite or multi phases in binary alloys or single austenite phase in high C and/or N alloys).^{4,7,12,13,17)} Considering a linked effects of applied stress and the strength of G.B. decohesion, the G.B. in high Mn austenite would be estimated to be intrinsically weak.

Finally, the data of 42Mn-5Cr are introduced for reference. As long as quenched into water after the solution treatment, this alloy fails in a ductile manner at 77 K: the enhancement of toughness and ductility

by slower cooling mentioned above conflicts with the usual understanding of heat treatments for high Mn austenitic steels like Hadfield steel.¹⁾ Slip lines were observed to be a little wavy, presumably owing to higher stacking fault energy caused by Cr and C additions. However, the difference in the slip lines is too subtle to conclude that the influence of Cr addition is ascribed to only the change in deformation manner, since the intergranular fracture in 40 Mn and 44 Mn occurs after a considerable amount of plastic flow.

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